IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

Applicants:	Robert J. Small and Zhefei J. Chen		
Title:	Compositions for Chemical-Mechanical Planarization of Noble-Metal-Featured Substrates, Associated Methods, and Substrates Produced By Such Methods		
Application No.:	10/057,206	Filing Date:	January 25, 2002
Examiner:	Michael Marcheschi	Group Art Unit:	1755
Docket No.:	CHEM.003US0	Conf. No.:	1702

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DECLARATION OF ROBERT J. SMALL AND ZHEFEI J. CHEN

Sir:

We, Robert J. Small and Zhefei J. Chen, declare under the penalty of perjury as set forth below.

1. We are joint inventors named in the above-identified patent application ("Application"). We were both employed by EKC Technology, Inc., the assignee ("Assignee") of the Application, when we invented subject matter disclosed in the Application that is now the subject matter of Claims 77-109 that are pending in the Application. A copy of Claims 77-109 is appended hereto in Appendix 1.

2. As part of our work for EKC, we developed compositions for polishing a substrate having a noble metal material, or a material comprising a noble metal, on its surface. The compositions comprised periodic acid, or H_5IO_6, which we often referred to as "PIA" and which is referred to hereinafter as PIA, and an abrasive.

3. In certain embodiments, these components were present in the compositions in a combined amount to render the substrate surface substantially planar upon chemical-mechanical polishing of the substrate surface.

4. In certain embodiments, the periodic acid was present in the compositions in an amount from about 0.05 to about 0.3 moles/kilogram, or from about 0.075 to about 0.3 moles/kilogram, or from about 0.075 to about 0.175 moles/kilogram.

5. In certain embodiments, the composition had a pH of from about pH 1 to less than pH 2 or from above pH 5 to about pH 10.

6. In certain embodiments, the abrasive was present in the compositions in an amount of from about 0.2 to about 6 weight percent or from about 0.2 to about 4 weight percent.

7. We believe that the subject matter set forth above in items 2-6, as well as other subject matter set forth herein, was conceived and reduced to practice before December 17, 1999.

8. Pending claims 77-93 of the Application relate to the subject matter set forth above in items 2-5, the reduction to practice of which is described below in relation to claim 77.

9. Pending claims 94-109 of the Application relate to the subject matter set forth above in items 2 and 4-6, the reduction to practice of which is described below in relation to claim 94.

10. Appended hereto in Appendix 2 are copies of two pages from a cleanroom notebook of Zhefei J. Chen (unnecessary portions of which have been redacted) concerning the chemical-mechanical polishing of two wafers, one wafer having a noble metal material, iridium (Ir), and the other wafer having a noble metal oxide material, iridium oxide (IrO_2), on its surface. The chemical-mechanical polishing was carried out using a composition

comprising about 2 weight percent of a pure α-Al_2O_3 abrasive, which we often referred to as "CR1" and is referred to herein as CR1, and $0.3\,\overline{3}$ or about 0.3 mole/kilogram of PIA (0.1 mole of PIA in a 300g composition), and having a pH of about 0.5 to 1. Upon chemical-mechanical polishing of the wafers, the latter wafer was shown to be edge polished, shining, and uniform, which was considered a positive result. The content of the two pages was prepared before December 17, 1999.

11. Appended hereto in Appendix 3 is a copy of a page of a cleanroom notebook of Zhefei J. Chen (unnecessary portions of which have been redacted) concerning a chemical-mechanical polishing composition for polishing wafers, each having a material comprising a noble metal material on its surface. The composition, which we often referred to as "ZCX200" and is referred to herein as ZCX200, comprised about 2 weight percent of an alumina abrasive, the commercial product "CR-30" manufactured by Baikowski Chimie Co. of Annecy Cedex 9, France, which is referred to herein as CR30, and about 0.1 mole/kilogram of PIA. ZCX200 was prepared to have a pH of about 1.5, or a pH of about 6 to about 7.5, measured either by probe or meter, or by paper, usually brought about by the addition of tetramethylammonium hydroxide (TMAH). The former is referred to herein as ZCX200 (1.5) and the latter is referred to herein as ZCX200 (6+). The content of the page was prepared before December 17, 1999.

12. Appended hereto in Appendix 4 is a copy of a page that was appended to a page of a cleanroom notebook of Zhefei J. Chen (unnecessary portions of which have been redacted) concerning the chemical-mechanical polishing of a number of wafers, each having iridium (Ir) on its surface. For three of the wafers, the chemical-mechanical polishing was carried out using ZCX200 (1.5) (the reference to a pH of $\leq$2.5 was used to indicate the

expected pH, while the parenthetic reference to a pH of 1.5 was used to indicate the actual pH, as measured), and for another of the wafers, the chemical-mechanical polishing was carried out using ZCX200 (6+). The chemical-mechanical polishing of each of the wafers was associated with a removal rate of 288, 400, 375, and 324 Å/min, respectively, each of which was considered a positive result. The content of the page was prepared before December 17, 1999.

13. The page mentioned above in item 12 also concerned the chemical-mechanical polishing of three wafers, each having Ir on its surface. The chemical-mechanical polishing was carried out using a composition comprising about 2 weight percent of CR30 abrasive and about 0.2 mole/kilogram of PIA, and having a pH of about 1.5 (the reference to a pH of $\leq$2.5 was used to indicate the expected pH, while the parenthetic reference to a pH of 1.5 was used to indicate the actual pH, as measured). The chemical-mechanical polishing of each of the wafers was associated with a removal rate of 250, 275, and 350 Å/min, respectively, each of which was considered a positive result. The content of the page was prepared before December 17, 1999.

14. The page mentioned above in item 12 also concerned the chemical-mechanical polishing of another wafer having Ir on its surface. The chemical-mechanical polishing was carried out using a composition comprising about 4 weight percent of CR30 abrasive and about 0.1 mole/kilogram of PIA, and having a pH of about 1.5 (the reference to a pH of $\leq$2.5 was used to indicate the expected pH, while the parenthetic reference to a pH of 1.5 was used to indicate the actual pH, as measured). The chemical-mechanical polishing of the wafer was associated with a removal rate of 290 Å/min, which was considered a positive result. The content of the page was prepared before December 17, 1999.

15. Appended hereto in Appendix 5 is a copy of a page of a cleanroom notebook of Zhefei J. Chen (unnecessary portions of which have been redacted) concerning the chemical-mechanical polishing of a number of wafers, each having Ir on its surface. For four of the wafers, the chemical-mechanical polishing was carried out using ZCX200 (6+), for which the pH was 7, as measured by meter, and 7.75, as measured by paper. The chemical-mechanical polishing of each of the wafers was associated with a roughness data (RMS data) of 7.28, 6.68, 5.64, and 6.47 Å, respectively, each of which was considered a positive result. The content of the page was prepared before December 17, 1999.

16. Appended hereto in Appendix 6 is a copy of a page that was appended to a page of a cleanroom notebook of Zhefei J. Chen (unnecessary portions of which have been redacted) concerning the chemical-mechanical polishing of a number of wafers, each having Ir on its surface. For one of the wafers, the chemical-mechanical polishing was carried out using ZCX200 (6+); for another of the wafers, the chemical-mechanical polishing was carried out using a composition comprising 2 weight percent of CR30 abrasive and about 0.2 mole/kilogram of PIA, and having a pH of about 7; and for yet another of the wafers, the chemical-mechanical polishing was carried out using ZCX200 (1.5). The chemical-mechanical polishing of each of the wafers was associated with roughness data (RMS or Scratch data) as set forth in the appended page, respectively, each of which was considered a positive result. The content of the page was prepared before December 17, 1999.

17. Appended hereto in Appendix 7 is a copy of a page that was appended to a page of a cleanroom notebook of Zhefei J. Chen (unnecessary portions of which have been redacted) concerning the chemical-mechanical polishing of a number of wafers, each having Ir on its surface. For one of the wafers, the chemical-mechanical polishing was carried out

using ZCX200 (6+); for another of the wafers, the chemical-mechanical polishing was carried out using ZCX200 (1.5); for yet another three wafers the chemical-mechanical polishing was carried out using a composition like ZCX200 (1.5), although the CR30 abrasive was replaced with another abrasive, a pure α-Al_2O_3 abrasive, which we often referred to as "CR1" and is referred to herein as CR1, a commercial alumina abrasive "CR6" manufactured by Baikowski Chimie Co. of Annecy Cedex 9, France, which is referred to herein as CR6, or an abrasive, which we often referred to as "CR85" and is referred to herein as CR85. The chemical-mechanical polishing of each of the wafers was associated with a removal rate of 325, 288, 286, 233, and 116 Å/min, respectively, each of which was considered a positive result. The content of the page was prepared before December 17, 1999.

18. Appended hereto in Appendix 8 are two copies of a page that was appended to a page of a cleanroom notebook of Zhefei J. Chen (unnecessary portions of which have been redacted) concerning the chemical-mechanical polishing of a number of wafers, each having Ir on its surface. (One copy of the page shows the page as appended to the notebook, absent the portion of the page that extends beyond the notebook, while the other copy of the page shows the page as appended to the notebook, including the portion of the page that extends beyond the notebook.) For three of the wafers, the chemical-mechanical polishing was carried out using ZCX200 (6+); for another three of the wafers, the chemical-mechanical polishing was carried out using a composition similar to ZCX200 (6+), although a pH of 7 was achieved using ammonium hydroxide (NH_4OH) instead of TMAH, which we often referred to as "ZCX206" and is referred to herein as ZCX206; and for another two of the wafers, the chemical-mechanical polishing was carried out using a composition similar to ZCX206, but comprising either 4 or 6 weight percent CR30 abrasive, rather than 2 weight

percent CR30 abrasive. The chemical-mechanical polishing of each of the wafers was associated with an Ir removal rate of 175, 100, 100, 250, 150, <100, 180, and 180 Å/min, respectively, each of which was considered a positive result. The content of the page was prepared before December 17, 1999.

19. Appended hereto in Appendix 9 is a copy of a page of a cleanroom notebook of Zhefei J. Chen (unnecessary portions of which have been redacted) concerning a chemical-mechanical polishing composition for polishing wafers having Ir on its surface. The composition comprises PIA and 2 weight percent of an Al_2O_3 abrasive and is associated with various estimated removal rates. The page included a note regarding watching the PIA precipitation range when the PIA is titrated with NH_4OH, indicating that when the PIA concentration is at 0.1 mole/kilogram, the PIA solution is clear to a pH of 10, and when the PIA concentration is at 0.3 mole/kilogram, the PIA solution precipitated at a pH of 1.5. It was considered desirable to use a clear, unprecipitated PIA solution. The content of the page was prepared before December 17, 1999.

20. Appended hereto in Appendix 10 is a copy of a page that was appended to a page of a cleanroom notebook of Zhefei J. Chen (unnecessary portions of which have been redacted) concerning the chemical-mechanical polishing of a number of wafers, each having Ir on its surface. The chemical-mechanical polishing was carried out using ZCX206 and was associated with a removal rate of about 180, 190, 190, and 300 Å/min, respectively, each of which was considered a positive result. The content of the page was prepared before December 17, 1999.

21. Appended hereto in Appendix 11 are copies of two pages, one containing a portion of a page that was appended to the page, of a cleanroom notebook of Zhefei J. Chen

(unnecessary portions of which have been redacted) concerning the chemical-mechanical polishing of a wafer having platinum (Pt) on its surface. The chemical-mechanical polishing was carried out using a composition comprising about 2 weight percent of an abrasive, which we often referred to as "CMP3100" and is referred to herein as CMP3100, and about 0.2 mole/kilogram of PIA, and having a pH of about 6.9, and was associated with a removal rate of 53 Å/min, which was considered a positive result. The content of the pages was prepared before December 17, 1999.

22. Appended hereto in Appendix 12 is a copy of a page of a cleanroom notebook of Zhefei J. Chen (unnecessary portions of which have been redacted) concerning the chemical-mechanical polishing of a wafer having Pt on its surface. The page included notes regarding lowering the pH of the chemical-mechanical polishing composition to 1 or 2, and regarding use of TMAH to titrate PIA to a pH of 7 or even 12. The content of the page was prepared before December 17, 1999.

23. Appended hereto in Appendix 13 are copies of two pages of a cleanroom notebook of Zhefei J. Chen (unnecessary portions of which have been redacted) concerning the chemical-mechanical polishing of a number of wafers, each having Pt on its surface. For two sets of two wafers, the chemical-mechanical polishing was carried out using a composition comprising about 2 weight percent of CR30 abrasive, and about 0.1 mole/kilogram of PIA, and having a pH of about 1.6; for another wafer, the chemical-mechanical polishing was carried out using a composition comprising about 2 weight percent of CR30 abrasive, and about 0.1 mole/kilogram of PIA, and having a pH of about 7.3. The chemical-mechanical polishing was associated with removal rates of 119 and 143.3 Å/min for one of the sets of wafers, 451.5 and 435 Å/min for the other set of wafers, and 43 Å/min for

the one wafer, respectively, each of which was considered a positive result. The content of the pages was prepared before December 17, 1999.

24. The pages mentioned above in item 23 concerned the chemical-mechanical polishing of yet another two wafers, each having Pt on its surface. The chemical-mechanical polishing was carried out using a composition comprising two abrasives, about 2 weight percent of CR85 abrasive and about 8 weight percent of another abrasive, which we often referred to as "CMPX077" and is referred to herein as CMPX077, and about 0.1 mole/kilogram of PIA, and having a pH of about 1.6. The CMPX077 served as a suspending agent. The chemical-mechanical polishing was associated with removal rates of 91 and 73.1 Å/min, respectively, each of which was considered a positive result. The content of the pages was prepared before December 17, 1999.

25. Appended hereto in Appendix 14 is a copy of a page of a cleanroom notebook of Zhefei J. Chen (unnecessary portions of which have been redacted) concerning a chemical-mechanical polishing composition for polishing wafers, each having a noble metal on its surface. The chemical-mechanical polishing composition comprised about 6 weight percent of CR30 abrasive and an amount of another abrasive, a commercial alumina abrasive "Alumina-C" manufactured by Deguss-Huls AG, which is referred to herein as Alumina-C, and about 0.1 mole/kilogram of PIA, and had a pH of about 1.75. The Alumina-C served as a suspending agent. The chemical-mechanical polishing composition was kept overnight and evaluated as to its suspension characteristics, which were considered positive. The content of the page was prepared before December 17, 1999.

26. The above-mentioned pages were generated from chemical-mechanical polishing experiments implemented by Zhefei J. Chen with the assistance of scientists under her direct supervision before December 17, 1999.

27. The above-mentioned pages and similar information dated both before and after December 17, 1999 have been kept among the usual business records of the Assignee in the ordinary course of business.

28. In view of the foregoing, we believe the invention as reflected in claim 77 and at least claims 79-91 depending therefrom of the Application was conceived and reduced to practice before December 17, 1999.

29. In view of the foregoing, we believe the invention as reflected in claim 94 and at least claims 95-106 and 109 depending therefrom of the Application was conceived and reduced to practice before December 17, 1999.

30. Each of us, respectively, further declares that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishment by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code, and that such willful false statements may jeopardize the validity of the application or any patent issuing thereon.

Executed on the date set forth below.

Date	Robert J. Small
6/15/04	Zhefei Chen
Date	Zhefei J. Chen

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Executed on the date set forth below.

June 16, 2004
Date

Robert J. Small

Date

Zhefei J. Chen